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Potential of a Platinum Electrode at Low Partial Pressures of Hydrogen and Oxygen

Part 2 - An Improved Gas-Tight System with a Negligible Oxygen Leak

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Chemistry Division

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PREVIOUS REPORT IN THIS SERIES

"Potential of a Platinum Electrode at Low Partial Pressures of Hydrogen or Oxygen," T. B. Warner and S. Schuldiner, NRL Report 6244, April 15, 1965

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Abstract: A tight electrochemical system was constructed in which the O_2 pressure (P_{O_2}) above the cell solution was less than 10^{-9} atm. A comparison of the potentials obtained with this system with data from NRL Report 6244 shows that very small amounts of O_2 leaking into a closed system can have marked effects on potential behavior at low H_2 pressures (P_{H_2}). Reduction of the O_2 leak to negligible proportions showed that: (a) the Nernst equilibrium relation for the H^+/H_2 couple holds only for P_{H_2} in excess of 10^{-6} atm; (b) at P_{H_2} less than 10^{-6} atm, trace amounts of O_2, even in the presence of several orders of magnitude more H_2, acted as an electrode poison, causing a positive deviation from the theoretical Nernst behavior; (c) in O_2-free solution, at P_{H_2} below 10^{-6} atm, the potential remained at 0.18 v positive to the normal hydrogen electrode (N.H.E.) and was independent of P_{H_2}. The potential-determining reaction in this region may be an exchange of H^+ in solution with H atoms dermasorbed in the Pt. The potential *vs* O_2 partial pressure relation was essentially the same as found in previous work at this Laboratory. Residual hydrogen associated with Pt at potentials from 0.18 to 0.20 v did not react with oxygen.

INTRODUCTION

In Part 1 of this series (1), Warner and Schuldiner determined open-circuit rest potentials on bright Pt as a function of O_2 or H_2 partial pressures from 10^{-2} to 10^{-7} atm. The O_2 leakage (from air) into the cell was estimated to give a partial pressure of about 10^{-7} atm. The Nernst equilibrium relation for the H^+/H_2 couple held only at H_2 partial pressures above 10^{-4} atm. At H_2 pressures below this value, the measured potentials were positive to the theoretical Nernst values. No satisfactory explanation could be given for this deviation, but it was felt that the trace of O_2 leaking into the system might be the cause. Even though a correction for the O_2 leak, assuming complete reaction with hydrogen at the Pt surface, was made, trace amounts of O_2 could possibly have remained on the surface when the H_2 partial pressure was below 10^{-4} atm. This O_2 might then act as a poison for the H^+/H_2 exchange (even though the H_2 partial pressure was several orders of magnitude higher) and give potentials positives to the theoretical equilibrium values. To determine if such traces of O_2 could affect the potential at low H_2 pressures, it was necessary to improve the gas-tight system to reduce further the leakage of O_2 into the cell. This was done in three ways:

1. Improved purification of the He carrier gas (Fig. 1) using two columns filled with Meyer-Ronge catalyst (2) instead of one.

2. Construction of a more compact gas-purification and cell system.

NRL Problems C05-06 and C05-13; Projects SR 007-12-01-0809 and RR 001-01-43-4754. This is an interim report on one phase of the problem; work on this and other phases of the problem is continuing. Manuscript submitted March 21, 1966.

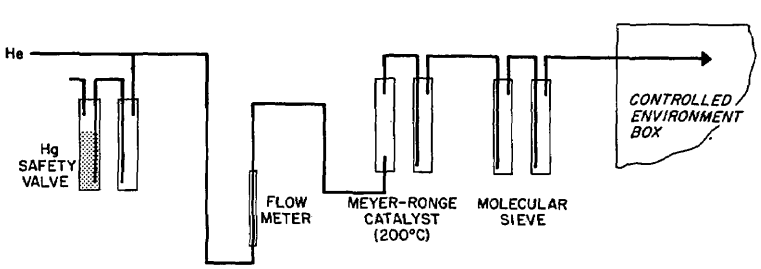

Fig. 1 — Helium-purification train outside of controlled-environment box

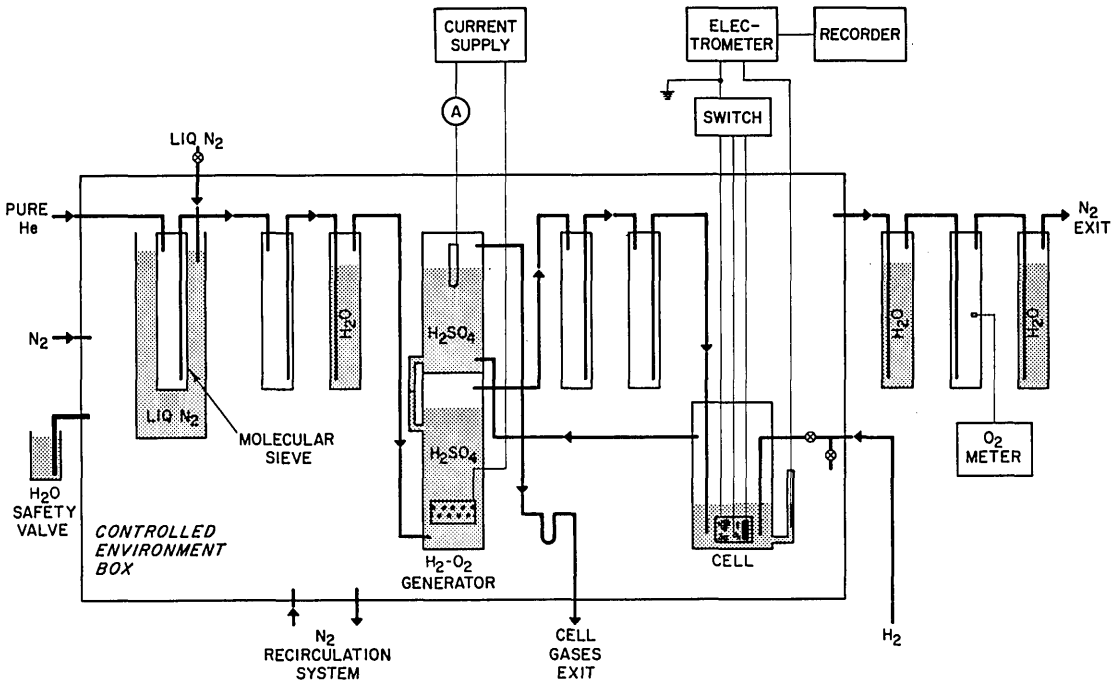

Fig. 2 — Electrochemical system inside of controlled-environment box and associated equipment

3. Installation of the critical part of the gas-purification system, the gas generator, and the cell in a controlled-environment box under an atmosphere of N_2 (Fig. 2). Thus the outside of the system was exposed to N_2 rather than air. The partial pressure of O_2 in the environment box was maintained at $(5 \pm 2) \times 10^{-4}$ atm. This meant that at the outside of the system the O_2 partial pressure was reduced to 1/400 of the value in air.

This improved gas-tight system reduced the O_2 leakage into the cell to immeasurable proportions, and, as the data will show, strongly affected the H_2 pressure-*vs*-potential relation.

EXPERIMENTAL CELL AND ASSOCIATED EQUIPMENT

Apparatus

The electrochemical cell (3) shown in Fig. 3 contained a miniature glass electrode, a Pt-wire electrode 5 cm long and 0.064 cm in diameter, a large Pt-gauze electrode with an area of about 100 cm^2, and a large tightly rolled cylinder of Pt gauze with a total geometric area of about 30 cm^2 platinized in leadfree platinic acid. The electrolyte was 1M H_2SO_4 maintained at (25 ± 2)°C.

The He-purification train is shown in Fig. 1. Copper tubing connected the He tank to the flow meter. From this point the system was glass, with one Viton-A O-ring connection between the two Meyer-Ronge catalyst columns. At the end of the second molecular-sieve column, a copper tube, gold-plated to prevent diffusion of H_2 in from the room atmosphere, was connected with a second Viton-A O-ring. This gold-plated copper tube conducted the purified He into the controlled-environment box and then on to the cold trap. Therefore, even though two Viton-A seals were used outside the box, one seal was between the two Meyer-Ronge columns, so any trace of O_2 which diffused through it was removed by the second Meyer-Ronge column. The second seal outside the box was in front of the molecular sieve-liquid N_2 cold trap, so that any O_2 which diffused through this O-ring would be frozen out. Since the liquid-N_2 cold trap was inside the controlled-environment box, diffusion of O_2 through any of the other seals was minimized. The manufacturer of the Viton-A O-rings claims

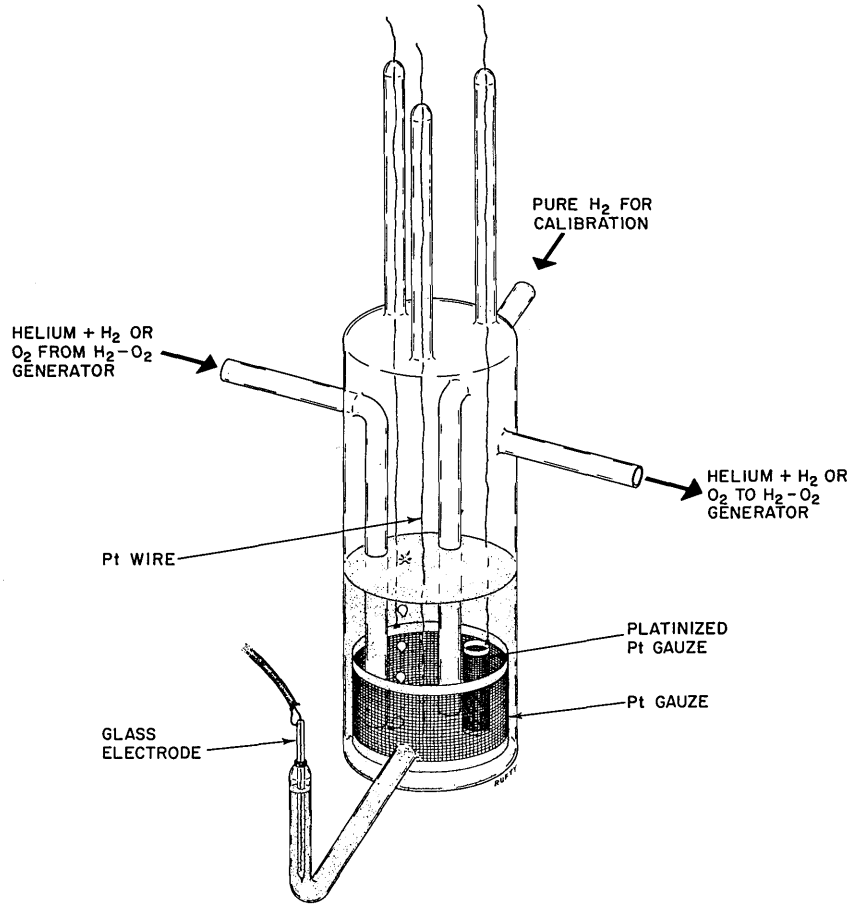

Fig. 3 — Representation of electrolytic cell. Glass pipe connections were actually used for electrode and gas connections and to connect upper and lower section of cell compartment. (See Fig. 1, p. 1143 of Ref. 3 for illustration showing construction of cell.)

a seal good to 10^{-6} torr, and previous work (1) indicated an oxygen leak in the entire system equivalent to about 10^{-7} atm, so with the new system it is felt that the O_2 leak within the cell should not exceed 10^{-9} atm. In any case, attempts to determine the amount of O_2 leaking into the cell from the atmosphere were fruitless because of its extremely low concentration.

The controlled-environment system with its electrolytic H_2 or O_2 generator (1), cell, and other components, is shown in Fig 2. The two stopcocks leading to the cell were Kern high-vacuum greaseless stopcocks with Viton-A diaphragms. The controlled-environment box was manufactured by Vacuum/Atmospheres Corporation and was 45 in. long by 30 in. deep by 33 in. high. The liquid N_2 cold trap consisted of a Linde 5A molecular sieve column in a Linde LD 10-l Dewar flask. The Dewar was filled via an insulated copper pipe that led outside the box where it was shut off with a valve. An N_2 atmosphere inside the box was obtained by passing tank N_2 into the box for several days, until the O_2 partial pressure inside the box dropped to about 10^{-3} atm. The flow of N_2 was then reduced, and the gas inside the box was recirculated through a bed of copper turnings at 450°C to remove O_2. Recirculation was via a high-speed gas pump with a flow of about 1.5 l/min. It was soon discovered that the evaporation of liquid N_2 maintained a sufficient gas flow through the box, and thus the tank N_2 was eliminated. Once the O_2 pressure dropped

to 5×10^{-4} atm, as determined on the outlet side with a Beckman Model E2 oxygen analyzer, recirculation of N_2 was no longer helpful in reducing the O_2 partial pressure. From that point on, the only sources of N_2 needed were from the evaporation of liquid N_2 and from the tank N_2 used to pump liquid N_2 into the Dewar. It was necessary to fill the Dewar every two weeks. This operation resulted in a large influx of N_2 into the box, so that a large outlet valve from the box had to be kept open to keep the internal pressure below 5 in. H_2O in excess of atmospheric pressure. In addition, an H_2O-filled safety valve was attached to the box. This valve would release N_2 when the pressure inside the box exceeded 5 in. H_2O above normal atmospheric pressure.

Electrical connections were made as shown in Fig. 2. A system of rotary switches was used to alternate every 5 min from one Pt electrode in the cell to another. The potential between each Pt electrode and the glass reference electrode was measured with a Keithley 610A electrometer and displayed on a Varian recorder.

Procedure

Glassware was cleaned with boiling nitric acid and rinsed 12 to 15 times with triply distilled H_2O (one stage of distillation from alkaline permanganate solution, another from H_2O containing fumed phosphoric acid, and the final two stages from a quartz still). The sulfuric acid solution was pre-electrolyzed for three days at 50 ma. This step was followed by calibration of the glass electrode against the Pt electrodes in H_2-saturated solution at 1 atm pressure. Hydrogen flow was then replaced with a flow of pure He always maintained at 40 std ml/min. Pressure within the cell was about 10 torr above atmospheric. The H_2 line was disconnected, flushed, and filled with He. The environment box was then sealed, and air was replaced with N_2 by the procedure described previously. Either H_2 or O_2 was introduced into the He carrier gas by a special electrolytic H_2 or O_2 generator designed at NRL (1). Gas-generation rates were calculated from constant electrolysis currents. The partial pressures were calculated from the rate of flow of He (known to 5 percent) and the rates of H_2 or O_2 generation (known to 1 percent).

The rate of attainment of steady-state potentials was very dependent on electrode area, especially at partial pressures below 10^{-6} atm. The platinized-Pt electrode area was so large that at low partial pressures it lagged far behind the other two Pt electrodes. All three electrodes would come to the same potential given sufficient time, but the platinized-Pt electrode required such a long time (months) at very low partial pressures that in most measurements only enough time was allowed for the Pt wire and gauze to come to the same steady-state values. The experiments which gave the data recorded in this paper took about 11 months. Several runs up and down the pressure scale were taken.

When pure He or He mixtures with low H_2 partial pressures flowed through the cell, the addition of liquid N_2 to the cold trap caused the Pt wire and gauze electrode potentials to deviate in a positive direction. Several hours were required for these electrodes to recover their original potentials. This effect is attributed to a trace of O_2 which was introduced during the cooling of the controlled-environment box, whose temperature usually decreased to about 17°C during the filling of the Dewar. This cooling caused a decrease in pressure in two arms of the cell, and the space above the solution in these compartments decreased as the cell solution rose slightly in them. This decrease may have caused a trace of O_2 trapped in these side arms or in the counter electrode side of the gas generator to go into solution, and/or it could have caused a further removal of traces of O_2 associated with the interior walls of the glass system. The rise of solution in these side arms showed that leaks did not exist. In any case, liquid N_2 was added only once every 14 days, so this effect was minimized.

EXPERIMENTAL RESULTS

The experimental results are shown in Fig. 4 for the hydrogen electrode and in Fig. 5 for the oxygen electrode. Values were obtained with both increasing and decreasing gas partial pressures and were taken only after steady-state potentials were reached. For H_2 partial pressures below 10^{-5} atm, steady-state meant no significant change in potential (values constant within ±3 mv, and no monotonic trend within this uncertainty) on

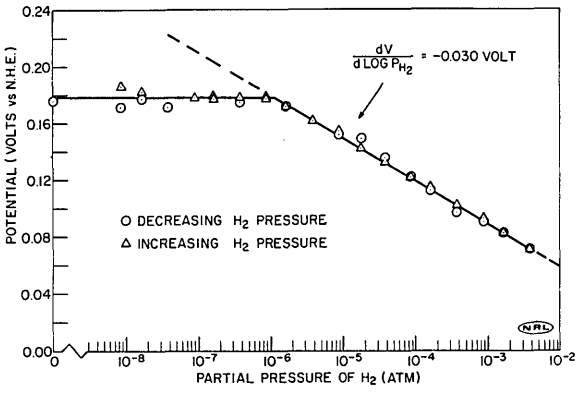

Fig. 4 — Relation between hydrogen partial pressure and rest potentials *versus* normal hydrogen electrode (N.H.E.). Dashed line shows normal Nernst potential relation for H^+/H_2 exchange.

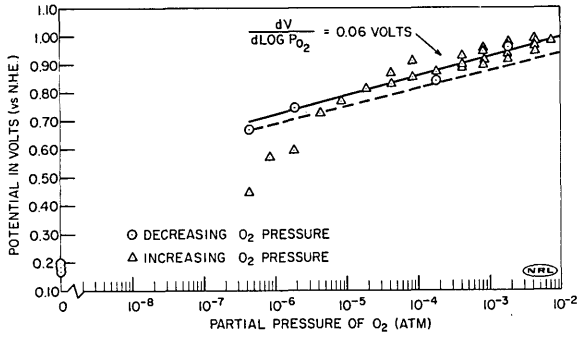

Fig. 5 — Relation between oxygen partial pressure and rest potentials. Dashed line indicates average values previously obtained at this Laboratory (1,3).

the Pt-wire electrode and gauze electrode for at least several days. The potentials of the two electrodes generally agreed to 2 mv, with occasional differences of 5 to 7 mv. For the platinized platinum gauze at the lowest partial pressures, the electrode continually changed in the expected direction, but at such a slow rate that it was not normally given time to reach the steady-state value of the other two electrodes.

For O_2 partial pressures, the rate of attainment of steady-state potentials for decreasing O_2 pressures was very slow, even for the Pt wire. Here also the potentials shown in Fig. 5 represent values that were unchanged for at least several days, but because of extreme system sluggishness, agreement between the two electrodes was poorer (5 to 30 mv), and random variations with time were larger. Also experiments were conducted in which the gas flow was changed from He containing first H_2 and then O_2, and *vice versa*. This reversal made no difference in the final rest potential if sufficient time was allowed for the rest potential to be attained.

In one experiment, after the electrodes were exposed to O_2-containing He for over two months, the O_2 flow was cut off. The rest potential on the Pt wire (0.2 v) was reached in about one week. Then, to test the purity of the solution, the cleanliness of the electrodes, and the tightness of the entire system, a single constant-current pulse was applied to the Pt wire in the He-saturated solution. This test took place six months after cleaning and closing the system. An anodic charging curve of the first applied current pulse gave an oscilloscopic trace that showed a small hydrogen region from 0.2 to 0.4 v, followed by double-layer and linear oxygen regions. The shape of the charging curve met published requirements (4) for a clean Pt surface free of detectable amounts of either oxidizable or unoxidizable organic contaminants or electrode poisons. The fact that H_2 was still associated with the Pt surface after such a long exposure to O_2 was unexpected, but it does account for the relatively low positive potentials of Pt observed in He-saturated solution.

DISCUSSION

Effect of Oxygen Leak

In order to estimate the maximum possible amount of O_2 that leaked into the cell, the following experiment was carried out. After passing pure He through the cell for five days, the average potential of the three Pt electrodes was 0.175 v. The sequence of changing the partial pressure of either O_2 or H_2 in the cell to the values shown in Fig. 6 was then carried out. At the end of each time noted for the given partial pressure shown in Fig. 6, the potential of the Pt wire, the electrode with the fastest response, is shown. It should be noted that these are not the final rest potentials at these partial pressures. These results are for the purpose of demonstrating the effects of small alternate additions of O_2 and H_2 on the potential. As the data show, potentials are sensitive to O_2

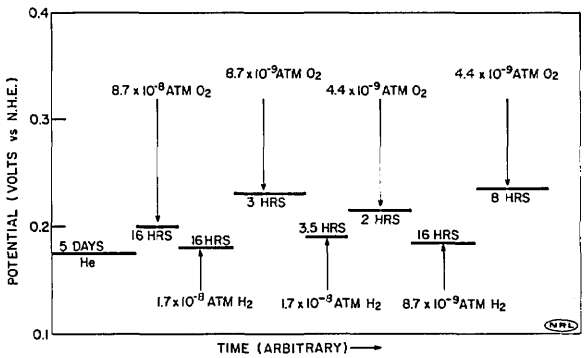

Fig. 6 — Effects of additions of small amounts of hydrogen or oxygen on potential of Pt wire

partial pressures as low as 4.4×10^{-9} atm and H_2 partial pressures as low as 8.7×10^{-9} atm. From this result we can conclude that the O_2 leak into the system cannot be, at most, more than enough to give a partial pressure of 10^{-9} atm. In fact, from the relatively fast response times shown in Fig. 6, it can be concluded that the O_2 leak, expressed in terms of resulting partial pressure, was probably under 10^{-10} atm.

Platinum Rest Potentials vs P_{H_2} or P_{O_2}

The data in Fig. 4 demonstrate that a normal Nernst equilibrium relation between potential and log P_{H_2} exists down to 10^{-6} atm H_2. At lower H_2 pressures the potential is independent of P_{H_2}, and this implies that the exchange-current density of the H^+/H_2 exchange at P_{H_2} below 10^{-6} atm is so low ($\approx 1.4 \times 10^{-9}$ A/cm², Ref. 5) that another potential-determining reaction takes over. Such a reaction could be

$$Pt-H \rightleftarrows Pt + H^+ + e,$$

where Pt—H either represents hydrogen atoms dermasorbed in the Pt skin or an alloy of Pt and H atoms. Schuldiner, Castellan, and Hoare (6) concluded that a similar potential-determining reaction took place on α Pd—H alloys.

Luk'yanycheva and Bagotskii (7) determined the initial open-current potentials of carefully degassed platinum in 1N H_2SO_4 degassed under vacuum. These potentials were 0.210 and 0.215 v. They observed that these rest potentials are close to the point of zero charge of Pt. The measurements in helium-saturated solution gave rest potentials 15 to 50 mv more negative than those of Luk'yanycheva and Bagotskii. In addition, the fact that a small amount of hydrogen was found to be associated with the electrode after prolonged exposure to oxygen showed that the rest potentials found in helium-saturated solutions and the plateau at 0.178 v (Fig. 4) are influenced by hydrogen.

Since the potential plateau in Fig. 4 is below the theoretical Nernst line, oxygen is not a factor, as it was in the first report of this series (1). The stability of Pt—H in this region is demonstrated by the rest potential in pure He-saturated solution. The experiments which showed that after an electrode has been in oxygen-rich He the rest potential in pure He is about the same indicate that this residual hydrogen does not react with O_2. In addition, the experiments showed that sorbed oxygen is in equilibrium with O_2 gas, because when the O_2 pressure drops to zero the O_2 coverage of the surface appears to drop to zero. The stability of this residual hydrogen indicates its irreversibility with respect to H_2; this conclusion supports the repeatedly stated view from NRL that this hydrogen is dermasorbed in the Pt skin and may be alloyed with Pt. The hydrogen content of the Pt electrode apparently did not change with H_2 partial pressures below 10^{-6} atm, as indicated by the unchanging electrode potential in this region. This fact may be contrasted with the dependence of hydrogen content of α Pd—H on H_2 partial pressure, as shown by Moon (8). However, much smaller amounts of H are involved than in the case of α Pd—H alloys.

One may argue that the plateau shown in Fig. 4 is due to an organic or other trace material in solution. However, the cleanliness test made after the system was used for six months shows that this possibility is unlikely. In addition, the discovery of hydrogen associated with the Pt after long exposure to O_2 indicates that hydrogen determines the potential. Another experiment to show the effects of hydrogen on potential was made by determining the rest potential in pure He. This step was followed by addition of O_2 to give a more positive rest potential. This second step was followed in turn by the addition of less than 10^{-6} atm H_2, which then gave the same rest potentials shown in Fig. 4. Furthermore,

the total area of Pt in solution was so large that impurity levels, which were below the detectable limit of the cleanliness test (4), would be minute. So even the traces of O_2 or H_2 used would overcome their effects (as was demonstrated by the reported tests).

The possibility must also be considered that the 0.2-v open-circuit potentials under pure He could be due to leakage of H_2 into the gas stream from the Pd-tube counter electrode in the electrolytic gas generator (1). The design of the gas generator made this improbable. Additional evidence that this did not occur came from the following experiment. After the electrodes were exposed to a He + O_2 mixture, pure He was substituted. While the potentials were changing to less positive values, the electrolyte contact with the Pd counter electrode in the gas generator was broken. The rate of potential change on the Pt electrodes in the cell did not change significantly, and final rest potentials were the same as previously found.

The rest potentials shown in Fig. 5 give essentially the same dependence of potential on O_2 partial pressure as was previously determined (1,3). There was, however, a significant difference. The broken line in Fig. 5 represents the average data previously found (1,3), and as can be seen, the data from this investigation indicated the same slope but an upward displacement of the average curve by about 40 mv. The reason for this difference is unknown. Stirring effects, effect of platinized Pt, effects of light, *etc.*, were all checked, and none seemed to be important. It also should be noted that the low potential values found at increasing very low O_2 partial pressures is most probably due to the very long times required to attain steady state. Decreasing values of O_2 partial pressures fell on the straight line shown in Fig. 5.

A current-voltage relation on the Pt wire was determined at $P_{O_2} = 4.36 \times 10^{-7}$ atm (Fig. 7). The open-circuit potential was a little above the rest potential later found. The linear region through zero count indicates that the potential is determined by a reversible reaction involving oxygen. In previous work (1,3), it was suggested that the potential was determined by an O_2/HO_2 exchange. The results of the NRL work confirm this suggestion.

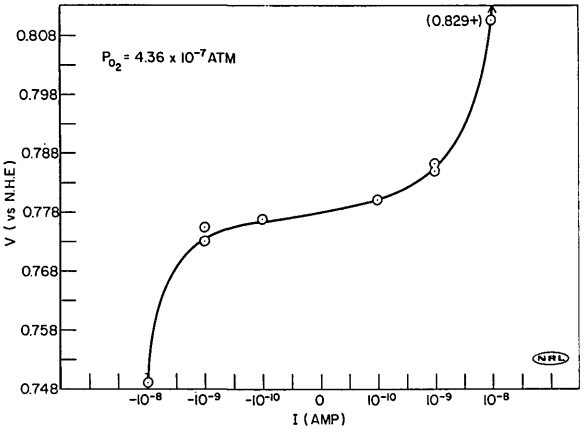

Fig. 7 — Anodic and cathodic polarization of Pt wire electrode

CONCLUSIONS

1. True steady-state open-circuit potentials can be obtained and maintained when sufficient steps are taken to insure solution and gas purity and electrode cleanliness.

2. Considerable periods of time (up to a month or more) are required to establish steady-state potentials with low partial pressures (below 10^{-4} atm) of H_2 or O_2. Response times are faster with hydrogen.

3. At low partial pressures, the surface area becomes dominating in the time for establishment of steady-state potentials. At higher partial pressures, the surface/volume ratio of the electrode is the determining factor.

4. The O_2 background in this improved system is less than 10^{-9} atm.

5. The deviation from Nernst behavior previously reported (1) for the H_2 electrode was due to the effects of small amounts of O_2 poisoning the H_2 equilibrium.

6. Below H_2 partial pressures of 10^{-6} atm, the steady-state potential is independent of P_{H_2}; *i.e.*, the Nernst potential relation for the H^+/H_2 equilibrium is not valid below 10^{-6} atm.

7. The potential plateau observed at 0.18 v for H_2 partial pressures below 10^{-6} atm is not due to an organic impurity.

8. The 0.18-v potential is believed to be established by an equilibrium involving H^+ ions in solution and hydrogen atoms dermasorbed in the electrode (or alloyed with the Pt). This reaction

has an exchange current density on the order of 10^{-9} amp/cm^2.

9. The residual hydrogen associated with Pt at potentials from 0.18 to 0.20 v did not react with oxygen.

10. The potential-O_2 partial-pressure relation found had the same slope as previously reported (1,3), although potentials at the same partial pressures ran about 40 mv higher.

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13. ABSTRACT

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	ROLE	WT	ROLE	WT	ROLE	WT
Hydrogen electrode Oxygen electrode Nernst potential relation Gas electrode thermodynamics Electrodes Platinum Gas-tight system Electrochemistry Electrode poisons Purification.						

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